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DICTIONARY FILE UPDATES: 23 NOV 2009 HIGHEST RN 1193434-24-0

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<http://www.cas.org/support/stngen/stndoc/properties.html>

=> s 826-62-0/rn

L5 1 826-62-0/RN

=> d

THE ESTIMATED COST FOR THIS REQUEST IS 2.05 U.S. DOLLARS  
DO YOU WANT TO CONTINUE WITH THIS REQUEST? (Y)/N:y

L5 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2009 ACS on STN

RN 826-62-0 REGISTRY

ED Entered STN: 16 Nov 1984

CN 4,7-Methanoisobenzofuran-1,3-dione, 3a,4,7,7a-tetrahydro- (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN 5-Norbornene-2,3-dicarboxylic anhydride (6CI, 8CI)

OTHER NAMES:

CN 2-Norbornene-5,6-dicarboxylic anhydride

CN 3,6-Endomethylenephthalic anhydride, 1,2,3,6-tetrahydro-

CN 3,6-Endomethylenetetrahydrophthalic anhydride

CN 3,6-Methano-4-cyclohexene-1,2-dicarboxylic acid anhydride

CN 3,6-Methylene-1,2,3,6-tetrahydrophthalic anhydride

CN 4-Oxatricyclo[5.2.1.0<sup>2,6</sup>]dec-8-ene-3,5-dione

CN 5-Norbornene-2,3-dicarboxylic acid anhydride

CN Bicyclo[2.2.1]hept-5-ene-2,3-dicarboxylic anhydride

CN cis-3,6-Endomethylene-1,2,3,6-tetrahydrophthalic anhydride

CN Endomethylenetetrahydrophthalic anhydride

CN HIMIC

CN NSC 3999

DR 66075-60-3

MF C9 H8 O3

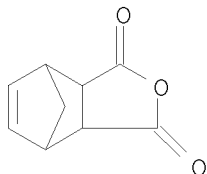
CI COM

LC STN Files: BEILSTEIN\*, CA, CAPLUS, CASREACT, CHEMCATS, CHEMINFORMRX,  
CHEMLIST, CSCHEM, CSNB, GMELIN\*, IFICDB, IFIPAT, IFIUDB, MSDS-OHS,  
RTECS\*, SPECINFO, TOXCENTER, USPAT2, USPATFULL, USPATOLD  
(\*File contains numerically searchable property data)

Other Sources: DSL\*\*, EINECS\*\*, TSCA\*\*

(\*\*Enter CHEMLIST File for up-to-date regulatory information)

10/923,271



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

752 REFERENCES IN FILE CA (1907 TO DATE)  
215 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA  
754 REFERENCES IN FILE CAPLUS (1907 TO DATE)

=> file caplus  
COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
3.01	64.85

FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE	TOTAL
ENTRY	SESSION
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FILE COVERS 1907 - 25 Nov 2009 VOL 151 ISS 22  
FILE LAST UPDATED: 24 Nov 2009 (20091124/ED)  
REVISED CLASS FIELDS (/NCL) LAST RELOADED: Aug 2009  
USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Aug 2009

CAPLUS now includes complete International Patent Classification (IPC) reclassification data for the third quarter of 2009.

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This file contains CAS Registry Numbers for easy and accurate substance identification.

10/923,271

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=> s 826-62-0/prep
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      4888293 PREP/RL
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      (826-62-0 (L) PREP/RL)
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L7      2 826-62-0/PUR
      (826-62-0 (L) PUR/RL)
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L9      145 L8 AND PY<2004
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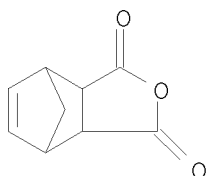
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      45378 ENDO
L10     7 L9 AND EXO AND ENDO
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THE ESTIMATED COST FOR THIS REQUEST IS 39.48 U.S. DOLLARS
DO YOU WANT TO CONTINUE WITH THIS REQUEST? (Y)/N:y
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L10 ANSWER 1 OF 7 CAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 2003:204619 CAPLUS
DOCUMENT NUMBER: 140:4929
TITLE: Stereochemical study of imides prepared by reaction of
primary amines with
bicyclo[2.2.1]heptene-5,6-dicarboxylic anhydride
AUTHOR(S): El Idrissi, M.; Amechrouq, A.
CORPORATE SOURCE: Departement de Chimie, Faculte des Sciences, UFR :
Chimie Organique, Meknes, Morocco
SOURCE: Physical & Chemical News (2002), 6(1),
126-129
CODEN: PCNHBU; ISSN: 1114-3800
PUBLISHER: Best Edition
DOCUMENT TYPE: Journal
LANGUAGE: French
OTHER SOURCE(S): CASREACT 140:4929
AB Imides are prepared in good yields by condensation of primary amine with
dicarboxylic anhydride at heat in acetic acid. A study of stereochem. by
spectroscopic anal. (IR, 1H and C13 NMR) shows that the imides are
obtained under two diastereoisomers forms - endo and exo
. The action of o-phenylenediamine on dioxo imides in the solvent acetic
acid/ethanol leads to benzimidazoles in good yields.
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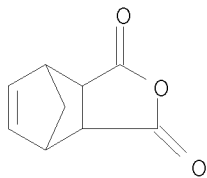
10/923,271

IT 826-62-0P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP  
(Preparation); RACT (Reactant or reagent)  
(stereochem. study of imides prepared by reaction of primary amines with  
bicyclo[2.2.1]heptene-5,6-dicarboxylic anhydride)  
RN 826-62-0 CAPLUS  
CN 4,7-Methanoisobenzofuran-1,3-dione, 3a,4,7,7a-tetrahydro- (CA INDEX NAME)



OS.CITING REF COUNT: 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD  
(1 CITINGS)  
REFERENCE COUNT: 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS  
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 2 OF 7 CAPLUS COPYRIGHT 2009 ACS on STN  
ACCESSION NUMBER: 1982:122270 CAPLUS  
DOCUMENT NUMBER: 96:122270  
ORIGINAL REFERENCE NO.: 96:20061a,20064a  
TITLE: Retro-Diels-Alder cleavage of endo  
-bicyclo[2.2.1]hept-5-en-2-ol  
AUTHOR(S): Prasad, J. V. N. Vara; Iyer, Padmini; Pillai, C. N.  
CORPORATE SOURCE: Dep. Chem., Indian Inst. Technol., Madras, 600 036,  
India  
SOURCE: Journal of Organic Chemistry (1982), 47(7),  
1380-1  
CODEN: JOCEAH; ISSN: 0022-3263  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 96:122270  
AB endo-Bicyclo[2.2.1]hept-5-en-2-ol (I) on refluxing with PhMgBr  
in Et2O gave HOCHMePh 40-50% arising from a retro-Diels-Alder cleavage of  
I into cyclopentadiene (II) and MeCHO. The retro-Diels-Alder reaction  
also took place when I was refluxed with MgBr2 or when the Na salt of I  
was refluxed; II and MeCHO could be isolated under these conditions. The  
exo-isomer of I was unaffected under similar conditions.  
IT 826-62-0P  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)  
RN 826-62-0 CAPLUS  
CN 4,7-Methanoisobenzofuran-1,3-dione, 3a,4,7,7a-tetrahydro- (CA INDEX NAME)



L10 ANSWER 3 OF 7 CAPLUS COPYRIGHT 2009 ACS on STN

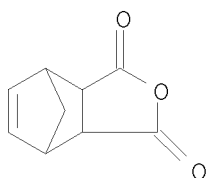
ACCESSION NUMBER: 1969:517440 CAPLUS  
 DOCUMENT NUMBER: 71:117440  
 ORIGINAL REFERENCE NO.: 71:21873a,21876a  
 TITLE: Structure of 5-norbornene-2,3-exo-dicarboxylic anhydride  
 AUTHOR(S): Destro, Riccardo; Filippini, G.; Gramaccioli, Carlo M.; Simonetta, Massimo  
 CORPORATE SOURCE: Univ. Milano, Milan, Italy  
 SOURCE: Tetrahedron Letters (1969), (38), 3223-6  
 CODEN: TELEAY; ISSN: 0040-4039  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English

AB Crystals of the title anhydride (I) are orthorhombic, space group P212121,  $a$   $7.985 \pm 0.002$ ,  $b$   $7.630 \pm 0.005$ ,  $c$   $12.770 \pm 0.002$  Å,  $d$  (exptl.) 1.42, with  $Z = 4$ , as measured by Cohen back reflection method using Cu K $\alpha$  radiation at 21°. Orientation with respect to crystallographic axes was obtained by angular scanning of the 3-dimensional Patterson synthesis. The correct solution of the structure was confirmed in the subsequent refinement which gave  $R$  0.098 for 596 observed independent reflections collected about the  $c$  axis. Anisotropic temperature factors for C and O atoms were used in the final least-sqs. refinement. The mol. geometry of I is quite similar to that of the endo compound. Bond distances and angles are similar to those in the endo compound. The dihedral angle between the anhydride group and the plane defined by atoms C-1, C-2, C-3, C-4 is 117°. In the norbornene nucleus the atoms C-1, C-4, C-5, C-6 are coplanar within less than 0.01 Å; the atoms C-1, C-2, C-3, C-4 are also coplanar within 0.01 Å.

IT 826-62-0P  
 RL: SPN (Synthetic preparation); PRP (Properties); PREP (Preparation)  
 (Structure of 5-norbornene-2,3-exo-dicarboxylic anhydride)

RN 826-62-0 CAPLUS

CN 4,7-Methanoisobenzofuran-1,3-dione, 3a,4,7,7a-tetrahydro- (CA INDEX NAME)



L10 ANSWER 4 OF 7 CAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1966:75509 CAPLUS  
 DOCUMENT NUMBER: 64:75509  
 ORIGINAL REFERENCE NO.: 64:14105a-d  
 TITLE: Photo-addition reactions of dimethyl maleate and dimethyl acetylenedicarboxylate with norbornene  
 AUTHOR(S): Hara, M.; Odaira, Y.; Tsutsumi, S.  
 CORPORATE SOURCE: Univ. Osaka, Japan  
 SOURCE: Tetrahedron (1966), 22(1), 95-100  
 CODEN: TETRAB; ISSN: 0040-4020

DOCUMENT TYPE: Journal

LANGUAGE: English

GI For diagram(s), see printed CA Issue.

AB Irradiation of a solution of norbornene in dimethyl maleate, with a 450-w. high pressure Hg lamp, at room temperature under N for 110 hrs. yielded exo-3,4-dicarbomethoxy-cis,-trans,cis-tricyclo[4.2.1.02.5]nonane (I), m. 82°, in almost quant. yield, based on the reacted norbornene. I was hydrolyzed by 10% NaOH to the 3,4-dicarboxylic acid (II), m. 202.5°, in 100% yield. The di-Ag salt of II was prepared in 90% yield, and was treated with Br in CCl<sub>4</sub> at -25° to form 3,4-dibromotricyclo[4.2.1.02.5]nonane (IV), m. 74-6°. IV was debrominated with Zn to give tricyclo[4.2.1.02.5]non-3-ene (V), a clear oil. Ozonolysis of V followed by reduction with LiAlH<sub>4</sub> gave exo-2,3-bis(hydroxymethyl)norbornane (VI), b<sub>l</sub> 132°, n<sub>30.5D</sub> 1.4982; 3,5-dinitrobenzoate m. 168-9°. For identification of the product, samples of endo-, exo-, and trans-2,3-bis(hydroxymethyl)norbornane were synthesized. The b.ps. of these compds. are: endo, 180-3°/30 mm. (m. 86°); exo, 110-16°/0.5 mm.; trans, 119-20°/0.3 mm. The m.ps. of their 3,5-dinitrobenzoates in the same order are: 180°, 169-70°, and 120°. Photolysis of norbornene in dimethyl acetylenedicarboxylate gave exo-3,4-dicarbomethoxytricyclo-[4.2.1.02.5]non-3-ene (VII), m. 185.5°. Hydrogenation of VII gave II. Ir, N.M.R., and uv spectra were used in determining the structure of the products. In the photoreaction in which I is formed, no homodimers of norbornene or of dimethyl maleate were detected. The photoreactions do not go by the charge transfer complex.

IT 826-62-0P, 5-Norbornene-2,3-dicarboxylic anhydride, exo

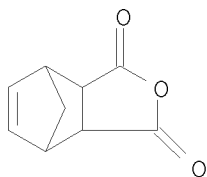
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RL: PREP (Preparation)

(preparation of)

RN 826-62-0 CAPLUS

CN 4,7-Methanoisobenzofuran-1,3-dione, 3a,4,7,7a-tetrahydro- (CA INDEX NAME)



OS.CITING REF COUNT: 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD  
(1 CITINGS)

L10 ANSWER 5 OF 7 CAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1963:26890 CAPLUS

DOCUMENT NUMBER: 58:26890

ORIGINAL REFERENCE NO.: 58:4437d-e

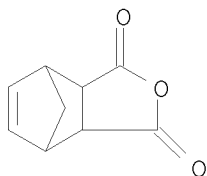
TITLE: Stereochemistry of cyclic compounds. XLV. Infrared spectra of the anhydrides of stereoisomeric cyclic ortho dicarboxylic acids

AUTHOR(S): Kucherov, V. F.; Serebryakov, E. P.; Kogan, G. A.

SOURCE: Zhurnal Obshchei Khimii (1962), 32, 760-5

CODEN: ZOKHA4; ISSN: 0044-460X

DOCUMENT TYPE: Journal  
 LANGUAGE: Unavailable  
 AB cf. CA 56, 428f; 58, 1371b; Cooke, CA 49, 9385c; Dauben and Epstein, CA 54, 5246g. Infrared spectra are reported for 24 alicyclic dicarboxylic anhydrides based on partly and completely hydrogenated systems of naphthalene, cyclopentanobenzene, phenanthrene, and 3,6-endomethylenephthalic anhydride. The unsatd. anhydrides with exo structure absorb at higher frequencies than do the endo analogs. Trans juncture of the anhydride ring with the hydrocarbon ring raises the frequency of the absorption band relative to the cis analog.  
 IT 826-62-0P, 5-Norbornene-2,3-dicarboxylic anhydride, exo  
 -  
 RL: PREP (Preparation)  
 (preparation of)  
 RN 826-62-0 CAPLUS  
 CN 4,7-Methanoisobenzofuran-1,3-dione, 3a,4,7,7a-tetrahydro- (CA INDEX NAME)



L10 ANSWER 6 OF 7 CAPLUS COPYRIGHT 2009 ACS on STN  
 ACCESSION NUMBER: 1955:64663 CAPLUS  
 DOCUMENT NUMBER: 49:64663  
 ORIGINAL REFERENCE NO.: 49:12376f-i,12377a-i  
 TITLE: Diene syntheses. XLI. The synthesis of nortricyclic derivatives  
 AUTHOR(S): Alder, Kurt; Brochhagen, Franzkarl  
 CORPORATE SOURCE: Univ. Cologne a. Rhein, Germany  
 SOURCE: Chemische Berichte (1954), 87, 167-79  
 CODEN: CHBEAM; ISSN: 0009-2940  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Unavailable  
 GI For diagram(s), see printed CA Issue.  
 AB cf. C.A. 49, 3858b. Condensation of 46.5 g. chloromaleic anhydride with cyclopentadiene (I) according to Synerholm (C.A. 39, 4054.8) gives 50 g. endo-cis-2-chloro-(exo)-1,4-endomethylene-5-cyclohexene-2,3-dicarboxylic anhydride (II), m. 160°. Reduction of 2 g. II in 10 cc. AcOH with 4 g. Zn dust 14 hrs., dilution with H2O, and extraction with ether give endo-cis-1,4-endomethylene-5-cyclohexene-2,3-dicarboxylic acid anhydride (III), needles, m. 163° [hydrotriazole, m. 225° (decomposition)]. Treating 2 g. II in 15 cc. AcOH and 15 cc. H2O 24 hrs. with 1.6 g. Br in 15 cc. AcOH and concentrating the mixture in vacuo gives a crude product, m. 200°, which with CH2N2 yields the mono-Me ester of IVa or IVb (R = Br), m. 146°. Catalytic hydrogenation of II in AcOEt with PtO2 gives the dihydro acid (V), m. 230° (decomposition) (di-Me ester, prepared with CH2N2, m. 87°). Refluxing V 1 hr. with 10% NaOMe gives 1,4-endomethylene-2-cyclohexene-2,3-dicarboxylic acid (Va), m.

212°. Boiling 10 g. II in 25 cc. H<sub>2</sub>O 10 min. gives VI, m. 204°, also obtained when II is dissolved in alkali and the solution is acidified and extracted with ether [Me ester (CH<sub>2</sub>N<sub>2</sub>), clusters of needles, m. 75°; acid chloride, obtained by refluxing the ester with SOCl<sub>2</sub> 4 hrs., b<sub>0.09</sub> 128-32°, m. 78°]. Adding slowly 9 g. bromomaleic anhydride in 15 cc. ether to 6 g. I in 25 cc. ether and keeping the mixture 24 hrs. give 90% endo-cis-2-bromo(exo)-1,4-endomethylene-5-cyclohexene-2,3-dicarboxylic anhydride (VII), m. 138°. Reduction of VII with Zn in AcOH gives III. Treating VII with Br in AcOH as above gives the Br lactone carboxylic acid (VIII), m. 238° (decomposition) [Me ester (CH<sub>2</sub>N<sub>2</sub>), m. 92°]; from the mother liquor of VIII is isolated an isomeric acid (IX), m. 168° (Me ester, m. 156°). Reduction of VIII and IX with Zn in AcOH gives endo-cis-1,4-methylene-5-cyclohexene-2,3-dicarboxylic acid (X), m. 188° (decomposition). Catalytic hydrogenation of VII and boiling 2 g. of the hydrogenated product with 15 cc. H<sub>2</sub>O gives endo-cis-2-bromo-(exo)-1,4-endomethylene-2,3-cyclohexanedicarboxylic acid (XI), m. 238° (di-Me ester, needles, m. 89°). Treating VIII with Zn in AcOH gives endo-cis-1,4-endomethylene-2,3-cyclohexanedicarboxylic anhydride, m. 167°. Refluxing 2 g. XI with 20 cc. 10% NaOMe 1 hr., oxidizing the mixture with 4% KMnO<sub>4</sub> until the purple color persists, and acidifying the filtered solution gives 0.2-0.5 g. of the 2,3-nortricyclenedicarboxylic acid (XIa), m. 250° (Me ester, oil; dianilide, prepared via the dichloride, felted needles, m. 241-2°). Heating 10 g. VII with 25 cc. H<sub>2</sub>O gives 5-5.5 g. VI, m. 204°. Addition of 9 g. dibromomaleic anhydride to 8 g. I in ether according to Diels and Alder (C.A. 24, 2119) gives endo-cis-2,3-dibromo-(exo)-1,4-endomethylene-5-cyclohexene-2,3-dicarboxylic anhydride (XII), m. 188°. Boiling 0.5 g. XII 0.5 hr. with 5 cc. H<sub>2</sub>O gives 3-bromo-5-hydroxy-2,3-nortricyclenedicarboxylic acid 3,5-lactone (XIII), m. 265° (mono-Me ester, prisms, m. 123°). Shaking 0.5 g. XIII in 50 cc. H<sub>2</sub>O containing 2 g. Na<sub>2</sub>CO<sub>2</sub> with 2 g. Raney Ni 12 hrs. in a H atmospheric gives VI. Keeping 20 g. chlorofumaroyl chloride and 10 g. I in 60 cc. ether several hrs., evaporating the ether, and treating the residue with 150 cc. H<sub>2</sub>O at 60-70° give 19 g. trans-2-chloro-(endo)-1,4-endomethylene-5-cyclohexene-2,3-dicarboxylic acid (XIV), m. 185°, which (2 g.), reduced with 4 g. Zn in 10 cc. AcOH, gives the trans-isomer (XV) of X, m. 189°. Treating 1 g. XIV in 20 cc. H<sub>2</sub>O with Br gives XVI or XVII, m. 230° [mono-Me ester (CH<sub>2</sub>N<sub>2</sub>), leaflets, m. 114°]. Evaporation of the aqueous mother liquor of XVI or XVII gives an isomeric Br lactone carboxylic acid, m. 174° (Me ester, rods, m. 104°). Reduction of XVI or XVII with Zn-AcOH gives XV. Catalytic hydrogenation of XIV gives the dihydro acid (XVIII), m. 213°. Thermal decomposition of XVIII gives Va, m. 212°. Refluxing 1 g. XIV in 20 cc. 10% Na<sub>2</sub>CO<sub>3</sub> or 20 cc. 20% KOH 1 hr. gives VI. VI (4.5 g.) in 15 cc. HNO<sub>3</sub> kept 8 days at 20° gives 3 g. 5-oxo-2,3-nortricyclenedicarboxylic acid (XIX), m. 239°, which is not further changed when treated with fuming HNO<sub>3</sub>. Esterification of XIX with CH<sub>2</sub>N<sub>2</sub> and refluxing the ester 0.5 hr. with H<sub>2</sub>NCONHNH<sub>2</sub>.AcOH gives the di-Me ester semicarbazone, m. 219° (decomposition). Refluxing XIX 3 hrs. with Ac<sub>2</sub>O gives the acetoxy lactone carboxylic acid, m. 181° [mono-Me ester (CH<sub>2</sub>N<sub>2</sub>), rods, m. 118°]. Refluxing the Me ester of 1 g. XIX 4 hrs. with 10 cc. 10% NaOMe gives VI. Refluxing 40 g. XIX in 200 cc. (CH<sub>2</sub>OH)<sub>2</sub> 1.5 hrs. with 28 g. KOH and 20 cc. 80% N<sub>2</sub>H<sub>4</sub>.H<sub>2</sub>O, heating the mixture to 190-5°, and refluxing it 4 hrs. at 195° give 9

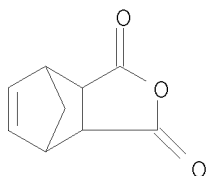
g. XIa [di-Me ester (XX) (MeOH-H<sub>2</sub>SO<sub>4</sub>)], b12 143°, n<sub>20D</sub> 1.4849, d<sub>20</sub> 1.1892]. Refluxing 35 g. XX in 250 cc. ether 0.5 hr. with 10 g. LiAlH<sub>4</sub> in 400 cc. ether gives 2,3-di(hydroxymethyl)nortricyclene, b0.08 101° [bis(3,5-di-nitrobenzoate), clusters of needles. m. 141°; bis(phenyl-urethan) m. 132°].

IT 826-62-0P, Bicyclo[2.2.1]hept-5-ene-2,3-dicarboxylic anhydride

RL: PREP (Preparation)  
(preparation of)

RN 826-62-0 CAPLUS

CN 4,7-Methanoisobenzofuran-1,3-dione, 3a,4,7,7a-tetrahydro- (CA INDEX NAME)



OS.CITING REF COUNT: 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD  
(1 CITINGS)

L10 ANSWER 7 OF 7 CAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1945:19036 CAPLUS

DOCUMENT NUMBER: 39:19036

ORIGINAL REFERENCE NO.: 39:2982d-i,2983a

TITLE: Unsaturated nitriles as dienophiles in the diene synthesis

AUTHOR(S): Blomquist, A. T.; Winslow, E. C.

SOURCE: Journal of Organic Chemistry (1945), 10, 149-58

CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 39:19036

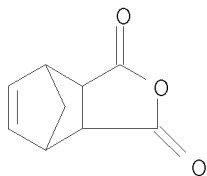
AB In an attempt to synthesize insecticides which possess the insecticidal properties of pyrethrum and of certain nitriles, the behavior of fumaronitrile (I), maleonitrile (II), and acetylenedicarbonitrile (III) as dienophiles in the Diels-Alder reaction with cyclopentadiene (IV) and diphenylfulvene (V) is studied. endo -cis-1,2,3,6-Tetrahydro-3,6-methanophthalic anhydride (VI), m. 164°, is prepared according to D. and A. (C.A. 22, 1144). trans-1,2,3,6-Tetrahydro-3,6-methanophthaloyl chloride (VII), b11 114-18°, is prepared in 83% yield according to A., et al. (C.A. 29, 2158.4). trans-Di-Me 1,2,3,6-tetrahydro-3,6-methanophthalate (VIII), b4 119-20°, m. 37-9°, is prepared in 80% yield by refluxing VI with absolute MeOH for 15 h. and passing dry HCl into the mixture for 5 h., and in 90% yield by reaction of MeOH with VII at 5°. When VIII is treated with concentrated NH<sub>4</sub>OH, trans-1,2,3,6-tetrahydro-3,6-methanophthalamide, m. 253-6° (decomposition), is formed. When 9 cc. IV is added to 7.8 g. I, b. 95-6°, in 50 cc. EtOH at 0° (reaction temperature should not exceed 35°), 92% trans-1,2,3,6-tetrahydro-3,6-methanophthalonitrile (IX), m. 95.5-6°, is obtained. Addition of II, m. 30-1°, prepared by heating a mixture of 11.4 g. maleamide, 50 g. P<sub>2</sub>O<sub>5</sub>, and 25 g. sea sand in a

vacuum, to IV gives 94% endo  
 -cis-1,2,3,6-tetrahydro-3,6-methanophthalonitrile (X), m. 155-6°.  
 Maleimide prepared according to Rinkes (C.A. 23, 5163) and IV give 90%  
 endocis-1,2,3,6-tetrahydro-3,6-methanophthalimide (XI), m. 184-5°.  
 XI is also prepared in 84% yield by refluxing a mixture of 21.6 g. NH<sub>4</sub>  
 cis-1,2,3,6-tetrahydro-3,6-methanophthalate with 7 cc. Ac<sub>2</sub>O for 2 h.  
 Hydrogenation of X with Pd catalyst gives 80%  
 cis-hexahydro-3,6-methanophthalonitrile, m. 145.5-6°; the trans  
 derivative prepared from IX m. 120-1°. Addition of III, prepared according to  
 Moureu and Bongrand (C.A. 14, 2335) from acetylenedicarboxamide, m.  
 290-2°, and P<sub>2</sub>O<sub>5</sub>, to IV gives 83%  
 3,6-dihydro-3,6-methanophthalonitrile (XII), m. 44-5°. An attempt  
 to prepare XII by dehydration of 3,6-dihydro-3,6-methanophthalamide (XIII)  
 failed. XIII, m. 211-12°, is obtained in 83% yield by addition of 25  
 g. di-Me acetylenedicarboxylate to 16.4 cc. IV and treatment of the  
 reaction product with concentrated NH<sub>4</sub>OH. Addition of I and V gives 25%  
 trans-1,2,3,6-tetrahydro-3,6-benzohydrylidenemethanophthalonitrile, m.  
 142-2.5° (decomposition). II and V give 46% exo  
 -cis-1,2,3,6-tetrahydro-3,6-benzohydrylidenemethanophthalonitrile, m.  
 172-3° (decomposition). III and V give 61%  
 3,6-dihydro-3,6-benzohydrylidenemethanophthalonitrile, yellow crystals  
 from EtOH, m. 168-9°. Resolution of  
 trans-1,2,3,6-tetrahydro-3,6-methanophthalic acid, m. 186-7°, is  
 carried out by 27 crystns. of its brucine salt, giving the pure active  
 acid, m. 166-8°, [ $\alpha$ ]<sub>D</sub><sup>20</sup> 89° (Me<sub>2</sub>CO), which confirms  
 the trans structure. Preliminary tests of the various nitriles derived  
 from XII indicate that some are effective as contact poisons for certain  
 species of insects. Those nitriles which contain a  $\Delta^4$ -double bond  
 appear to be the most active.

IT 826-62-0P, Bicyclo[2.2.1]-5-heptene-2,3-dicarboxylic anhydride  
 RL: PREP (Preparation)  
 (preparation of)

RN 826-62-0 CAPLUS

CN 4,7-Methanoisobenzofuran-1,3-dione, 3a,4,7,7a-tetrahydro- (CA INDEX NAME)



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 (6 CITINGS)